Preparation of $MgAl_2O_4$ Films on Si by Single Precursor OMCVD

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Heterometallic alkoxides have recently gained renewed interest as the source materials for ceramics and other materials. The use of monomeric heterometallic alkoxides in particular, as precursors for organometallic chemical vapor deposition (OMCVD) is increasing due to their relatively low melting points as well as their fixed ratios of constituent metal atoms.

Thin films of $MgAl_2O_4$ can be used as insulator in SOI (silicon on insulator) structure or as buffer layer for high temperature superconductors, and are usually prepared by chemical vapor deposition (CVD). The conventional CVD method, however, requires high deposition temperatures and suffers from the difficulty in controlling and obtaining correct film compositions using many source materials. Therefore OMCVD method and the development of the appropriate precursors are being studied more often than before.

In this investigation the single precursors $Mg[Al(O^iPr)_x(O^tBu)_{4-x}]_2$, where x = 1 or 2, and $MgAl_2(O^tBu)_8$ were synthesized and employed in preparing $MgAl_2O_4$ films on silicon substrates.

A 1:2 mixture of magnesium and aluminum isopropoxide with a small amount of $HgCl_2$ was sealed in a three-neck flask to which purified ^{t}BuOH/benzene and a small amount of CCl_4 was added by the Schlenk technique, and this was refluxed for 48 hours obtaining a gray-colored solution. Refluxing was continued until the resulting isopropyl alcohol was effectively removed by forming an azeotropic mixture. The remaining solvent was then evaporated, and after drying, a solid chunk much like a piece of soap was obtained. This, when purified by sublimation, yielded white crystalline substance. Proton NMR, IR, and mass spectroscopy revealed it to be a mixture of $Mg[Al(O^iPr)_x(O^tBu)_{4-x}]_2$, where x = 1 or 2.

In another experiment, aluminum was made to dissolve in ^{t}BuOH/benzene using a small amount of $HgCl_2$, and after adding magnesium, the solution was refluxed for 48 hours to obatin a dark red solution. Solvent evaporation followed by sublimation produced white crystalline substance the structure of which has yet to be determined but most probably is $MgAl_2(O^tBu)_8$. This compound was readily soluble in organic solvents and was stable in nitrogen atmosphere up to about 300 ^{0}C.

Deposition of the films were carried out in a simple Pyrex vacuum system built in this laboratory. The silicon substrates were cut to the size 2 x 1 cm^2 from a *p* type Si(100) wafer with thickness of 0.25 inch. The deposition temperature was varied between 500 ^{0}C and 800 ^{0}C. Heating of the substrate was accomplished by resistive method. The precursors were transported to the substrate by vaporization at 40 ^{0}C to 100 ^{0}C and a diffusion pumping system. Films deposited were analyzed with SEM, XRD, XPS, and Raman spectroscopy and found to be $MgAl_2O_4$. Although more work still has to be done, this result confirms the usefulness of $Mg[Al(O^iPr)_x(O^tBu)_{4-x}]_2$ and $MgAl_2(O^tBu)_8$ as the precursors of $MgAl_2O_4$ films.